Validation Worksheets - ICP

Site:_____ QC Level:_____ Project No.:_____

SDG#_____ No. Samples_____ Matrix_____ Lab:_____

Attach Copy of Case narrative, Lab Sample ID pages, and Flagged Data Tables

Analysis Performed (check all that apply): ☐ SW-846 ☐ CLP ☐ 200.7 ☐ AES ☐ MS

Parameter	Criteria	Acceptable	Not Acceptable
Holding Time; Preservative	6 months to digestion/6 months to analysis HNO_3 to pH < 2 (H_2O) 4°C (± 2°C) (H_2O and soil)		
Initial Calibration:			
Requirement	All target analytes		
Levels	A blank and at least 5 calibration standards, one at or below the CRQL		
Frequency	Each time instrument is set up and after CCV failure		
Criteria	Correlation Coefficient ≥ 0.995 %D of all non-zero standards ± 30% of true value y-intercept of curve must be less than CRQL		
ICV/CCV Standards			
Level	**ICV** – Highest mixed calibration std. **CCV** – Mid level calibration std.		
Frequency	**ICV** – Immediately after initial calib. **CCV** – Every 2 hours, beginning and end of analytical run		
Criteria	90 – 110% recovery		
Blanks			
Level	No contaminants should be found in any blank.		
Frequency	**ICB** – immediately after ICV **CCB** – every 2 hours and immediately after CCV **Method Blank** – 1 per SDG or preparation batch, whichever is more frequent		
Criteria	**ICB** – No detections **CCB** – No detections **Method blank** – No detections		
Interference Check Sample			
Frequency	At the beginning of an analytical run after the ICV/ICB		

Parameter	Criteria	Acceptable	Not Acceptable
Criteria	+ CRQL or + 20% of true value (whichever is greater		
Laboratory Control Sample			
Level	Final digestate = 2 x CRDL conc.		
Frequency	1 per group of aqueous or soil samples in an SDG, or with each batch of samples digested, whichever is more frequent.		
Criteria	70 – 130%R for all except Sb and Ag Sb and Ag must fall within 50 – 150%R		
Duplicate Samples			
Level	Cannot use blank or PE sample		
Frequency	1 DUP per matrix per SDG		
Criteria	**Aqueous:** 20% RPD when both samples > 5 x CRQL CRQL when 1 or both samples are < 5 x CRQL		
	Soil: 35% RPD when both samples > 5 x CRQL 2 x CRQL when 1 or both samples are < 5 x CRQL		
Matrix Spike Samples			
Level	Cannot use blank or PE sample		
Frequency	1 MS per matrix per SDG or 5%		
Criteria	75 – 125%R when original result is < 4 x concentration of spike added.		
Post-digestion Spike Samples			
Level	Cannot use blank or PE sample. Spiked at 2 x CRQL.		
Frequency	Run when MS outside 75-125%R and original result is < 4 x concentration of spike added.		
Criteria	75 – 125%R		
ICP Serial Dilution			
Level	Cannot use blank or PE sample.		
Frequency	1 per matrix or SDG, whichever is more frequent		
Criteria	**Aqueous:** %D between original and 5 fold dilution < 10% where orig. conc. > 50 x MDL **Soil:** %D between original and 5 fold dilution < 15% where orig. conc. > 50 x MDL		
ICP-MS Tune Analysis			
Level	100 µg/L of Be, Mg, Co, In, and Pb		

Parameter	Criteria	Acceptable	Not Acceptable
Frequency	Tuning solution analyzed 5 times prior to instrument calibration.		
Criteria	%RSD of 5 runs < 5%		
MS Internal Standards			
Level	5 of the following: Li^6 isotope, Sc, T, Rh, Tb, Ho, Lu, and Bi. If Li^6 isotope is used, Li^6 enriched standard must be used.		
Frequency	Every sample must contain		
Criteria	% Relative Intensity = 60 – 125% of the response in the calibration blank.		

Level IV/D/Definitive Data with Raw Data

Note: calculations/transcriptions may be written down in raw data package or on separate paper as proof that they were performed.

YES	NO	
☐	☐	Transcriptions checked for 10% of the data (raw vs. summaries)?
☐	☐	Quantitation calculations checked for 10% of ALL data (i.e. calibrations, check standards, spikes, dups, results)?

Reviewers Signature:

_____ Date____/____/____